XPS Case Study #3

Chemical State Analysis of Aluminum Oxide

XPS analysis is a technique that enables the determination of the chemical states of elements present within the analyzed region (Φ10 - 100 µm, depth less than 10 nm) through detailed analysis of the peak positions detected from individual electron orbitals. In this study, the XPS spectrum of aluminum oxide was examined. As shown in Figure 1, the spectral shape reflected differences in chemical state (metallic or oxide). Therefore, the relative fractions of the chemical states were determined.

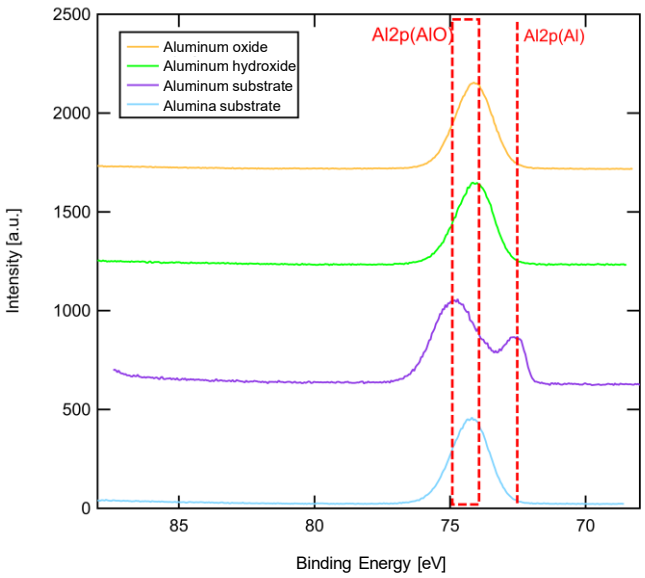


Fig.1 : Al2p spectra of each sample (normalized intensity)

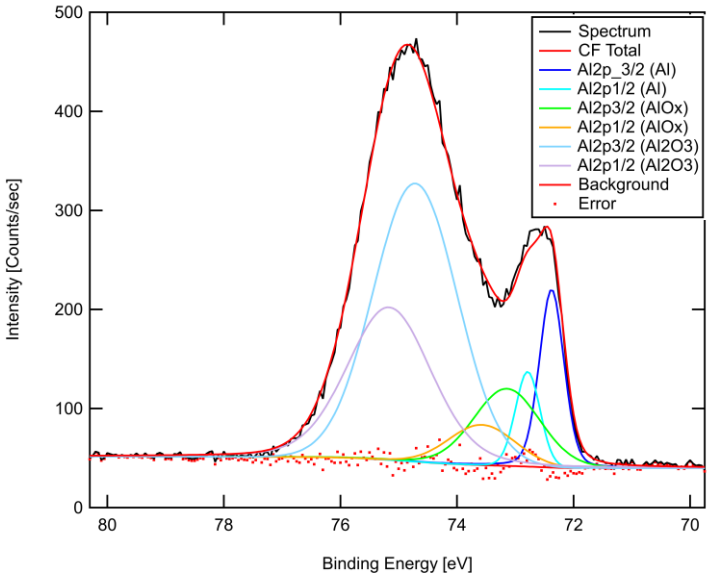


Fig.2 : Curve-fitting results for the Al2p spectrum of the Al substrate

Various aluminum oxide species, including Al_2O_3 and $Al(OH)_3$, may be present. However, their Al2p3/2 peak positions lie within the range of 74 – 75 eV, and it is often difficult to determine the individual chemical states by curve-fitting analysis. In such cases, evaluation is performed by determining the composition ratio x (or oxidation state) of the overall aluminum oxide (AlOx).
The evaluation is first performed using a survey spectrum to check whether any impurities are present. The detected elements are then measured using high-resolution spectra to enable detailed curve-fitting analysis. The measured spectra are analyzed to determine the bonding-state ratios, from which the elemental composition ratios are calculated (see table below).

The following four aluminum material samples were evaluated.

- Aluminum oxide(powder)
- Aluminum hydroxide(powder)
- Aluminum substrate
- Almina substrate

Sample	C1s	O1s			Al2p			Total	AlOx
			O bonded to C	O bonded to Al		Al metal	O bonded to Al		
Aluminum oxide	10.7	59.0	3.4	55.6	30.3	0.0	30.3	100.0	1.8
Aluminum hydroxide	39.1	45.1	4.9	40.2	15.8	0.0	15.8	100.0	2.5
Aluminum substrate	28.9	44.4	2.1	42.3	26.7	5.7	21.0	100.0	2.0
Almina substrate	24.6	49.0	4.8	44.2	26.4	0.0	26.4	100.0	1.7

Table 1: Analysis Results of the Aluminum Oxide Composition Ratio (x) for 4 Samples (The unit is atom%)

Based on these results, the amounts of Al bonded to O and O bonded to Al were determined, and the composition ratio was calculated. As a result of the analysis, aluminum oxides were considered to exist primarily in two forms : aluminum oxide (x = 1.5) and aluminum hydroxide (x = 3.0). Although the two powder samples were affected by the outermost surface layer, the calculated compositions were close to the stoichiometric ratios. The results suggest that the alumina substrate contains oxide species similar to those present in the aluminum oxide powder, whereas the oxide film on the aluminum substrate contains oxide species that include aluminum hydroxide.
In this analysis, the bonding states of the Aluminum substrate were evaluated as a combination of two components: stoichiometric aluminum oxide and aluminum oxide with an intermediate composition. By further considering differences in the composition ratios of these oxide species, the effects of different surface treatments on the same Al substrate can be evaluated in greater detail.

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